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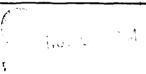
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report summarizes ONR research on the development and use of spectroscopic techniques for the study of electrochemical interfaces and electrode processes occurring at such interfaces. A number of in-situ techniques have been used including ultraviolet-visible reflectance spectroscopy and ellipsometry, infrared attenuated total reflectance spectroscopy, surface enhanced and resonant Raman spectroscopy, and Mossbauer spectroscopy. These in-situ techniques have provided important insight into the electronics and the

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properties of adsorbed molecules, ions and atoms at electrochemical interfaces. They have also been used to examine the adsorption isotherms and kinetics of adsorption-desorption.

The <u>in-situ</u> techniques, however, are insufficient to establish the type of adsorption sites or compositional questions. For this reason substantial use has also been made of <u>ex-situ</u> surface physics techniques including LEED, AES, XPS and secondary ion mass spectroscopy. Special hardware and techniques have been developed as part of the ONR contract research to facilitate the transfer of electrodes between the electrochemical and ultrahigh vacuum environments and <u>vice versa</u> with the possibility for restructuring and contamination at a <u>minimum</u>. The report describes the application of these techniques to adsorption on single crystal electrodes.

### FINAL REPORT

### submitted to the

# OFFICE OF NAVAL RESEARCH

### for research on

### ELECTROCHEMICAL INTERFACES AND ELECTRODE PROCESSES

Contractor: Case Western Reserve University, Cleveland, Ohio 44106

Department:

Case Center for Electrochemical Sciences and

the Chemistry Department

Contract No.:

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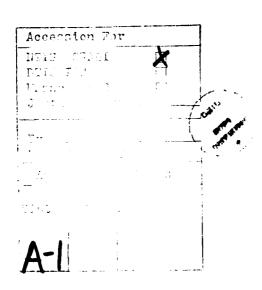
Termination Date:

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Project Director:

Ernest Yeager, Hovorka Professor of Chemistry

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### T. RESEARCH OBJECTIVES AND OVERVIEW

The objectives of the ONR sponsored research are two-fold:

- the understanding of the factors controlling electrosorption of various species on metal and semiconductor electrodes;
- 2. the relationship of the kinetics of various electrode processes to the electrode properties and adsorption on electrodes.

While electrochemical methods have high sensitivity to adsorption on electrodes (down to a small fraction of a monolayer), they lack sufficient atomic level specificity to identify uniquely the type of sites, the molecular configuration of the adsorbate, the nature of the bonding to the surface, the interaction between adsorbed species and the competitive aspects with respect to solvent adsorption. Consequently much of the emphasis on this ONR sponsored research has been in the use of complementary techniques to study adsorption on electrodes. The <u>in situ</u> techniques which have been used for this purpose as part of the ONR research and the systems which have been studied have included

- 1. ultraviolet-visible reflectance spectroscopy automatic ellipsometry
- 2. infrared attenuated reflectance spectroscopy
- 3. surface enhanced and resonant Raman
- 4. Mossbauer spectroscopy

These measurements have yielded critically needed information concerning the electronic and vibrational properties of electrosorbed species and anodic films as well as the adsorption isotherms and the kinetics of the adsorption-desorption and surface rearrangements. They are insufficient by themselves to pin down the type of adsorption sites or compositional questions. For this reason, we have also made substantial use of ex situ

techniques to complement the electrochemical and <u>in situ</u> spectroscopic measurements. These have included

- 1. LEED
- 2. Auger electron spectroscopy
- 3. X-ray photoelectron spectroscopy
- 4. secondary ion mass spectroscopy

Special apparatus and techniques have been developed as part of the ONR contract research to facilitate the transfer between the electrochemical and ultrahigh vacuum interface and vice versa with the possibility of restructuring minimized. The types and distribution of adsorption sites is dependent on the surface topography and particularly the crystallographic plane. LEED and ultrahigh vacuum techniques have proved essential for the preparation of single crystal surfaces and their characterization before and after the electrochemical measurements. While many groups are carrying out electrochemical measurements on single crystals, their work is in general not indicative of surfaces with known zone axes. To prepare a clean surface corresponding to a given surface plane requires in general vacuum-LEED techniques and very special transfer procedures. Furthermore, the surfaces usually reconstruct during the electrochemical measurements and post-electrochemical LEED examination is essential. The effort to carry out such "ultra" electrochemical experiments, however, is very demanding and time-consuming.

The electrochemical systems to which such <u>in situ</u> and <u>ex situ</u> methods have been applied in this ONR sponsored research have included

 underpotential deposited metals on gold: UV-visible, ellipsometry, LEED, AES (1-4,26)

- 2. electrosorbed hydrogen on platinum: LEED, AES, XPS (5-8,26).
- 3. adsorbed halogen ions on Au: UV-visible reflectance (including kinetics electroreflectance admittance) (9).
- 4. adsorbed transition metal macrocyclic complexes on Ag, Au, Pt, basal plane of graphite, high area carbon: UV-visible reflectance Raman, Mossbauer (Fe, Co<sup>57</sup>) (10-14,22,27).
- 5. adsorbed probe molecules on Ag, Pt, carbon, semiconductors: Raman (15-18
- 6. anodic films and passivation layers on Pt, Au, Fe, Fe-Cr, Ni, Ag: UV-visible reflectance, ellipsometry, infrared, Raman, Mossbauer, XPS, AES, SIMS (19-25,27,28).

This research for the most part is summarized in various technical reports and publications. More recent developments over the past year are summarized in the following sections of this report.

### II. RECENT DEVELOPMENTS IN CONTRACT RESEARCH

A. Upgrading of the thin-layer-electrochemical-cell-LEED-Auger-XPS system

The thin-layer-electrochemical cell-LEED system was designed and constructed in the latter 1970's as part of the ONR contract research. This system permits single crystal electrode surfaces to be prepared and characterized with LEED in ultra-high vacuum and then transferred into a thin layer electrochemical cell without removing the electrode from the vacuum chamber. The electrochemical measurements are carried out in the presence of ultrapure argon as a supporting gas. Following the electrochemical measurements, the electrode surface is again returned to the ultra-high vacuum and reexamined with LEED and AES. Most of the recent studies have been carried out with dilute aqueous HF since this electrolyte is not specifically adsorbed over a substantial range with single crystal surfaces such as gold

<sup>\*</sup>Some of the more recent measurements on iron and the iron-chromium alloys have been carried out as part of the ONR/SRO project at Case on the passivation of iron and ferrous alloys from Sept. 1979 - August 1982.

and platinum. The system was used to examine the electrosorption of hydrogen on single crystal platinum and underpotential deposition of lead on single crystal gold. The results have shown that each of the low index planes of these face-centered cubic single crystal metals have quite different adsorption characteristics. On the basis of these measurements it is clear that fundamental studies must be carried out on single crystal surfaces to be meaningful. On the other hand, the post-electrochemical LEED examination indicates that the surfaces often reconstruct in the electrochemical environment either giving new LEED patterns or becoming sufficiently disorganized so as not to give a pattern at low electron beam voltages where the diffraction arises principally from the surface layer. Only over relatively narrow potential ranges and then only on some crystal planes is it possible to maintain the original surface configuration. Furthermore, the AES measurements indicate that the surfaces are very easily contaminated and that even only a small fractional surface coverage can produce gross changes in the electrochemical properties.

On the basis of these earlier studies during 1978-81, the conclusion was reached that the study of electrochemical adsorption is only meaningful on single crystals for solid electrodes and that such electrochemical studies must be carried out with pre- and post-electrochemical LEED measurements with the most effective transfer possible. Consequently we have directed further effort to refining and upgrading this electrochemical-LEED-AES system. The changes made during the past one and a half years have included the following:

#### 1. XPS-AES capability

The original system had only the limited capability afforded by the Varian RFD grid LEED-AES system. A Vacuum Generators hemispherical electron analyzer (CLAM-100) has been added together with a dual target X-ray source (Al, Mg) and an electron gun. The available funds were insufficient to secure the signal processing computer system from Vacuum Generators. A surprisingly effective system, however, has been developed by our group using a Commodore Pet microprocessor together with a Nicolet signal average (1170) and associated Peripheral equipment. This system now provides us a high performance XPS-AES capability. A diagram of the overall system as it now stands is shown in Fig. 1.

### 2. Increased pumping speed

The vacuum system consists of two principal vacuum chambers:

<u>Chamber A</u>: This chamber contains the LEED-AES-XPS system plus a sputtering gun and electron beam heater. The vacuum is usually in the  $10^{-11}$  Torr range.

<u>Chamber B</u>: This chamber houses the thin layer electrochemical cell plus a quadrupole mass spectrometer. Ultra-pure argon is used in this chamber at ca. 0.3 atm when the thin-layer cell is formed. This chamber has heretofore been only pumped with a triode ion pump and after sufficient time reached the  $10^{-10}$  Torr range. This chamber is connected to Chamber A with an isolation value between the two chambers and a magnetically operated transfer wand to convey the single crystal electrode between the two chambers.

After the electrochemical measurements it is important to purge out the support gas (argon) and volatilize the electrolyte  $(0.1\underline{M}\ HF)$  as rapidly as possible. To accomplish such, a turbomolecular pump has been connected to Chamber B, which pulls the pressure down to  $10^{-8}\ Torr$  in 90 - 120 s following the completion of the electrochemical experiments. The sample is then transferred into Chamber A through the low conduction connection between the two chambers. In 3 to 5 minutes, the sample is again in position for LEED or AES-XPS examination at a pressure of  $10^{-10}\ Torr$ . This has greatly reduced the possibility of post-electrochemical contamination of single crystal surfaces such as Pt with CO and other materials released from the

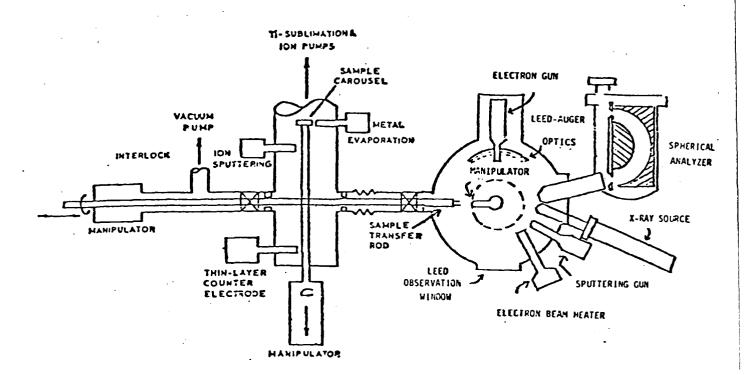


Fig. 1. LEED-Auger-ESCA-thin layer electrochemical cell system

walls of the chamber or present in the argon or released from the triode pump while pumping out the large amount of argon in Chamber B. With this system it has been possible to reduce the carbon level on Pt single crystal surfaces in the post-electrochemical AES measurements to 5 to 10% of a monolayer. Even so, we are still seeking to lower the carbon surface contamination in the post-electrochemical measurements to a much lower level  $\leq 2\%$ .

### 3. Electrolyte handling system

The electrolyte handling system has been completely rebuilt to provide better control of the purity of the electrolytic solutions such as  $0.1\underline{M}$  HF and better control of the volume delivered for the electrochemical measurements. The delivery system only exposes the electrolyte to Teflon. The portions of the Teflon delivery system outside the vacuum chamber are surrounded by an argon atmosphere to eliminate the possibility of  $0_2$  diffusing through the walls into the solution.

### 4. Thin-layer electrochemical cell

Heretofore the thin-layer cell has consisted of the single crystal working electrode and a combined counter-reference electrode of  $\alpha$ -Pd-H. This counter-reference electrode, however, can prove erratic under some circumstances. Consequently a three-electrode system has also been developed in which a Teflon-surrounded  $\alpha$ -Pd-H electrode with only the end exposed is used as a separate reference, placed between the working and counter electrodes. This arrangement is currently being tested and should result in a larger number of successful runs as well as more accurate voltammetry results.

The system for cleaning, charging and pretesting the  $\alpha$ -Pd-H counter electrode has also been greatly upgraded, using a third small vacuum chamber

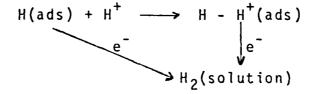
with its own ion pump.

B. Studies\_of the electrosorption of hydrogen on single crystal platinum surfaces.

In 1977-79, hydrogen adsorption and desorption were examined on the low index planes of Pt in  $H_2SO_4$  solutions using the earlier version of the thin-layer cell-LEED-Auger system. When the potential was cycled only in the 0.0 to 0.55 V vs.  $\alpha$ -Pd-H, the voltammetry curves on the (111) and (110) planes each showed only one major adsorption-desorption peak corresponding to weakly bound hydrogen and with a total charge only a small fraction of that expected for a monolayer. The (100) surface also showed only one predominant adsorption-desorption peak at more anodic potentials corresponding to more strongly bound hydrogen also with the charge corresponding to a small fraction of a monolayer. Cycling the potential into the anodic film region (1.45 V) resulted in the growth of these peaks on each of the surfaces. This could be explained in two ways: (1) the surfaces restructured at more anodic potentials and (2) carbon or other contamination was oxidized off the surface by cycling out to the anodic potentials, thus freeing up the surface for H adsorption. Both probably occurred. The use of sulfuric acid as the electrolyte, however, interfered with the postelectrochemical LEED and AES examination since this electrolyte is difficult to volatilize and becomes highly oxidizing as water is preferentially lost.

Consequently, the decision was made to repeat the measurements with 0.1M HF as the electrolyte using the partially upgraded system. More emphasis was placed on the purity of the low index surfaces before introduction into the electrochemical environment and the question of carbon

monoxide contamination during the pump down following the electrochemical measurements. The voltammetry results on the Pt 111 and 110 (2 x 1) surfaces were rather similar. Cycling into the anodic range resulted in the growth of the principal hydrogen peak. The subsequent LEED patterns for electrodes not cycled into the anodic film region indicated the patterns to be similar to those initially obtained although the background was increased. Carbon levels for the initial surfaces were usually below detection and in the postelectrochemical AES measurements were less than 10% monolayer level. When the electrode was cycled into the anodic film region (1.45 V), the hydrogen peaks again grew but the LEED showed major restructuring with loss of most of the pattern at low electron beam voltages. The voltammetry results on the Pt 100 (5 x 20) in HF, however, were quite different (see Fig. 2). With potential sweeps in the hydrogen adsorption and so-called anion region, the cathodic peak appeared in the anodic sweep.\* This cathodic spike is attributed to the irreversible adsorption of hydrogen attending a structural rearrangement of the surface. The hydrogen can be desorbed very irreversibly at 0.8 V. Furthermore, potential Window opening experments indicate that this hydrogen can be cathodically desorbed at potentials less than 0.15 V. The process is proposed to be



Dr. P. Ross has just reported that F. Wagner and himself at Lawrence Berkeley have now also observed such a cathodic spike in the anodic sweep for Pt(100).

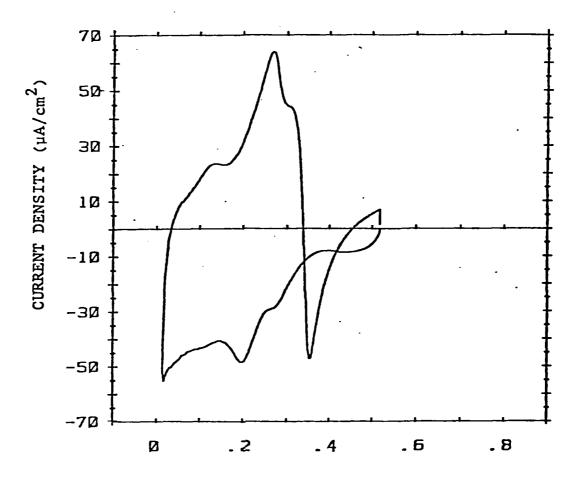


Fig. 2. Linear sweep voltammetry Pt (100) - 5 x 20 in 0.1M HF. Sweep rate = 50 mV/s.

which may occur in one or two steps. These correspond to the Heyrovsky and Horiuti mechanisms for  $\rm H_2$  generation.

Highly irreversible structural rearrangements attending the adsorption of hydrogen on the  $Pt(100 - 5 \times 20)$  surface from the gas phase have been reported by Norton et al. (28). The hydrogen is adsorbed at  $120^{\circ}$ K with the loss of the 5 x 20 overlayer and cannot be desorbed until 350-500°V. The cathodic peak in the anodic sweep at ca. 0.40 V in Fig. 2 is analogous to the low temperature adsorption from the gas phase and the desorption at the much more positive potential of 0.80 analogous to the high temperature desorption.

With multiple sweeps into the anodic film region (1.4 V) the cathodic spike is depressed and the voltammetry curve in the hydrogen region becomes similar to that found for  $0.05\underline{M}$  H<sub>2</sub>SO<sub>4</sub>. Post-electrochemical LEED examination of the Pt (100) surface indicates loss of the 5 x 20 overlayer even if the potential is swept only in the limited range 0 - 0.5 V. With sweeps well into the anodic film region, the spot pattern becomes difficult to discern with a large amount of background diffuse scattering. With higher electron beam voltages the LEED pattern again exhibits well-defined spots. The surface after exposure to quite positive potentials has undergone substantial rearrangement.

These studies of hydrogen electrosorption on Pt in  $0.1\underline{M}$  HF were carried out before the installation of the Vacuum Generators Clam 100 hemispherical analyzer and the dual target x-ray source for XPS measurements. We are presently in the process of repeating a few of the measurements in  $0.1\underline{M}$  HF with post-electrochemical studies of the surfaces with XPS and AES to check more carefully on the presence of trace surface impurities.

- During the period 1976 79, the LEED-AES thin layer electrochemical cell system was used to study the underpotential deposition of lead on various gold single crystal surfaces. This work is summarized in published papers [15 and a technical report (51)]. During the last year of this contract, research has been initiated on a thorough LEED study of underpotential deposited layers on single crystal surfaces using this apparatus after the upgrading described earlier in this report. Underpotential deposition of lead on the low index surfaces of silver has been chosen for this study. The work has been continued under a follow on ONR contract. Research also has been initiated on the simultaneous underpotential deposition of two species, for example lead and copper on gold. The initial work is on polycrystalline gold but research under the continuation of this porject will involve single crystal gold substrates.
- D. Studies of anodic film formation on platinum single crystal surfaces

  When the platinum single crystal surfaces are exposed to potentials
  in the anodic film region and withdrawn at potentials cathodic to this
  region, the LEED patterns indicate major surface changes. Work has been
  initiated to pin down the nature of these changes from the LEED patterns as
  functions of electron beam voltage. The platinum surfaces are also being
  withdrawn from the electrochemical cell at different potentials in the
  anodic film region to investigate the nature of the film with LEED and to
  check on the state of the platinum and adsorbed oxygen with XPS. In preliminary experiments with electrodes withdrawn in the anodic film region we have
  found fluorine in the XPS but not if the electrode is withdrawn at less
  positive potentials. This warrants careful investigation. This work is
  on-going and promises much needed information to understand the electrocatalytic properties of platinum for such reactions as O<sub>2</sub> electroreduction

Various transition metal macrocyclic complexes on electrode surfaces

Various transition metal macrocyclic complexes adsorbed on electrode
surfaces exhibit high catalytic activity for various electrode reactions.

The reduction of 0<sub>2</sub> on such surfaces has been of special interest. Some of
these complexes such as the iron tetrasulfonated phthalocyanines (TSPc)
have high activity for the overall 4-electron reduction of 0<sub>2</sub> to 0H<sup>-</sup> or H<sub>2</sub>0
while others such as the CoTSPc promote two-electron reduction of 0<sub>2</sub> to peroxide
(5,29,30). The former process is much preferred for such applications as
fuel cells, metal-air batteries and certain industrial electrolytic processes.

The state of these adsorbed complexes on electrode surfaces and their interactions with 0<sub>2</sub> and reactive intermediates such as 0<sub>2</sub>- and H0<sub>2</sub>- are not well understood. As part of the research sponsored by both DOE and ONR, the Raman and visible reflectance spectroscopy of adsorbed layers of Co and Fe-TSPc as well as transition metal-free H<sub>2</sub>TSPc have been examined in acid, neutral and alkaline electrolytes (10-13). The Raman measurements have been carrie out on silver electrodes roughened by cycling the potential. The reflectance of the adsorbed Co and Fe-TSPc species were examined on the basal plane of stress-annealed graphite, platinum and gold (10,13). The Raman and UV-visible spec of the solution phase Co- and Fe-TSPc were also examined with and without 0<sub>2</sub> present in acid, neutral and alkaline electrolytes. Unfortunately fluor-escence interfered with obtaining the Raman spectra of the H<sub>2</sub>-TSPcNa<sub>4</sub>, although the fluorescence was not pronounced for this species adsorbed on the electrode surface, probably because of quenching of the fluorescence by energy exchange with the metal (13).

The visible reflectance spectra of the adsorbed Co- and Fe-TSPc are similar to those of the solution and solid phase species. An easily detectable change occurs in the reflectance spectra with exposure of the

system to  $0_2$  at constant electrode potential. The Raman spectra for the adsorbed species have essentially the same frequencies as the solution phase and solid phase species. The relative intensities, however, are quite different and furthermore are strongly dependent on the electrode potential as well as laser excitation frequency. The peak heights fall into two distinct types of potential dependence. This suggests that the macrocyclic may be present on the surface in two orientations or configurations with the amount in each configuration dependent on potential. Unfortunately, the selection rules for molecules exhibiting both surface enhancement and intrinsic resonance or pre-resonance enhancement are not well identified and hence identification of the configuration on the surface is uncertain. The Raman also is not sensitive to the axial ligand using visible laser excitation (488.0, 514.5 or 632.8 nm) and it is necessary to carry out the Raman studies in the ultraviolet to pin down whether di-oxygen is bound in the axial position and if so in a superoxide or peroxide state. Further work is planned using ultraviolet Raman.

Some of the possible configurations for the Co- and Fe-TSPc adsorbed species are shown in Fig. 3. In view of the strong adsorption and similarity of the spectra to that of the solids, we favor configurations b and d.

Mossbauer effect spectroscopy provides a means for examining the Fe and Co macrocyclics including the Fe- and Co-Pc in high area carbon electrodes in porous carbon electrolyte structures (14,22,27). We have been successful in examining the Fe phthalocyanine and Co tetramethoxyphenyl porphyrin in situ in alkaline and acid electrolytes as well as ex situ. This is an exciting development since it is possible to gain fundamental insight into the behavior of the complexes within high area electrodes. These studies are in an early state but already quite useful information has been obtained. For the Fe

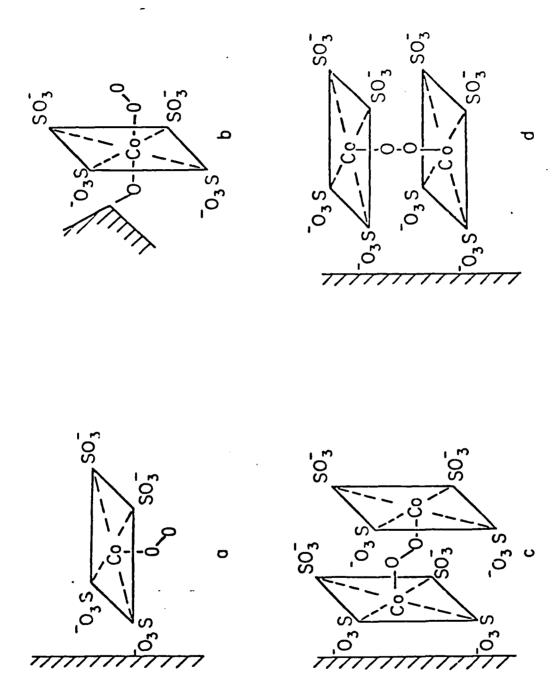


Fig. 3. Possible configurations for CoTSPc adsorbed on an electrode surface. [Kötz and Yeager (11)]

phthalocyanines, the concentration of the naturally present  $^{57}$ Fe is sufficient with the FePc catalyzed electrode used as the absorber for the  $\gamma$ -rays from a  $^{57}$ Co source. For the Co complexes, the species have been synthesized with  $^{57}$ Co and the electrodes loaded with this species used as the  $\gamma$ -ray emitter. The relaxational properties of the intermediate  $^{57}$ Fe produced from the  $^{57}$ Co are such that the system behaves similarly to Fe.

For the FePc in a moderately high area Teflon bonded carbon electrode, two sets of doublets are observed: one associated with the bulk solid and the other with the adsorbed species. The quadrupole splitting for the adsorbed species (0.88 mm/s) is low and provides evidence that the adsorbed species is probably bound through an axial 0 ligand to the electrode surface. This would be compatible with configuration b in Fig. 3.

The Mossbauer effect spectroscopy has also provided some interesting information on the effects of the electrode treatment on the state of the FePc in the electrode (27). For example, with FePc on carbon treated at  $\sim 400\,^{\circ}\text{C}$  in  $\text{H}_2$  before electrochemical examination, the Mossbauer measurements revealed the six doublets of metallic iron in a dispersed form. In helium under the same preparative conditions, there was no evidence of instability from the Mossbauer measurements. The Mossbauer measurements have also indicated that the amount of the macrocyclic complex in the adsorbed and bulk states in the electrode is quite sensitive to the preparative procedure.

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  - 48. Passivation of Iron, C. Chen, B. D. Cahan, D. J. Wheeler and E. Yeager, 6 February 1979.
  - 49. Optical and Electrochemical Studies of Adsorbed Transition Metal Complexes and Their O<sub>2</sub> Electrocatalytic Properties, E. Yeager, J. Zagal, B. Z. Nikolic and R. R. Adzic, 1 May 1979.
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- 4. "Electrode Surface Studies by LEED-Auger," W. E. O'Grady, M.Y.C. Woo, P. L. Hagans and E. Yeager, J. Vac. Sci. Technol. 14 (1977) 365-367.
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- 9. "Recent Advances in the Understanding of Electrocatalysis and its Interrelation to Surface Chemistry," E. Yeager, <u>ibid</u>., pp. 149-171.
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- 15. "Optical Studies of the Passivation of Iron," D. J. Wheeler, B. D. Cahan, C. T. Chen and E. Yeager, Proc. 4th International Symposium on Passivity, R. Frankenthal and J. Kruger, eds, The Electrochemical Society, Princeton, NJ. 1978, pp. 546-563.

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- 18. "Reflectance Spectra of Monolayers of Tetrasulfonated Transition Metal Phthalocyanines Adsorbed on Electrode Surfaces," B. Z. Nikolic, R. R. Adzic, and E. Yeager, J. Electroanal. Chem. 103 (1979) 281-287.
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- 32. "Oxygen Cathodes: Present Status and Problem Areas," by Ernest Yeager, Workshop on Renewable Fuels and Advanced Power Sources for Transportation, Boulder, CO, June 17 and 18, 1982, pp. 27-54.
- 33. "Study of Phthalocyanines in Aqueous Solutions and Adsorbed on Electrode Surfaces," by B. Simic-Glavaski, S. Zecevic, and E. Yeager, J. Electroanal. Chem., 150, 469-479 (1983).

# C. Papers Presented at Conferences and Meetings

- "LEED-Auger Studies of Electrode Surfaces," by W. O'Grady, M. Woo, P. Hagans and E. Yeager, The Electrochemical Society Natl. Meeting, Washington, May 1976.
- 2. "Ellinsometric Studies of the Tynamics of Iron Passivation," by B.D. Cahan, T. Greev, C.T. Chen, and E. Yeaner, ibid.
- 3. "Oxygen Electrocatalysts for Life Support Systems," by ". O'Grady, C. Iwakura and F. Yeager, Intersociety Conference on Environmental Systems, San Diego, Calif., July 1976.
- 4. "Structural Studies of Electrode Surfaces," by ". Algrady, ". Hoo, P. Hagans, and E. Yeager, International Society of Electrochemistry, International Meeting, Zurich, September 1976.
- 5. "Studies of the Dynamics of Passivation Using Automatic Fllipsometry," by B.P. Cahan, T. Greev, C.T. Chen and E. Yeager, ibid.
- 6. "Electrode Surface Studies by LEFD-Auger," by W. O'Grady, M. Woo, P. Hagans and E. Yeager, American Vacuum Society, "lational Symposium, Chicago, September 1976.

- 7. "Gas Fed Porous Electrodes," by E. Yeader and D. Cahan, The Electrochemical Society, Mational Meeting, Las Megas, October 1976.
- "Application of Electrode Kinetics Principles to Energy Conservation Problems," by E. Yeager, Workshop on Energy Conservation in Industrial Electrochemical Processes, \*rnonne "ational Laboratory, August 19-12, 1976.
- 9. "Electrochemical Mydrogen Adsorption on the Pt (111) and (100) Surfaces," by M. E. O'Grady, M.Y.C. Moo, P.L. Hagans and F. Yeager, The Electrochemical Society, Mational Meeting, Philadelphia, May 8-13, 1977.
- 10. 'Recent Advances in the Understanding of Flectrocatalysis and its Interrelation to Surface Chemistry," E. Yeager, <u>ibid</u>.
- "An Overview of the Electrochemical Interface and Ontical Spectroscopic Studies," <u>F. Yeager</u>, Colloquium on Ontical Properties of Solid-Liquid Interfaces, sponsored by the CMPS, La Colle sur Loup, France, "May 23-28, 1977.
- 12. "Electrochemical Properties of Flectrode Surfaces in Relation to Materials Research," E. Yeaner and M.F. O'Grady, Symposium on Materials from a Chemical Viewpoint, American Chemical Society, Mational Meeting, Mew Orleans, March 20-25, 1977.
- 13. "The Optimization of Oxygen Cathodes and Anodes for Various Applications," E. Yeager, Gordon Research Conference on Flectrochemistry, Santa Barbara, Calif., January 17-21, 1977.
- 14. "Transient Ellipsometric Studies of the Growth and Reduction of Passive Films on Iron," C.T. Chen, B.D. Cahan, and E. Yeager, National Meeting, The Electrochemical Society, Seattle, May 21-26, 1978. (Extended Abstract 61).
- 15. "Paman Spectra of Molecules Adsorbed on Flectrodes," G. Hagan, B. Simic-Glavaski, and E. Yeaner, ibid. (Extended Abstract 535).
- 16. "Ellipsometric Spectroscopy of the Passive Film on Iron," <u>B.D. Cahan</u>, C.T. Chen and E. Yeaner, International Meeting of the International Society of Electrochemists, Budanest, 26 August 2 September, 1973, (Extended Abstract 426-427.)
- 17. "The Raman Spectroscopy of Adsorbed Species on Flectrode Surfaces,"

  13. Hagen, B. Simic-Glavaski, and <u>E. Yeager</u>, <u>ibid</u>. (Extended Abstract 441-3).
- 18. Optical and Electrochemical Studies of the Passivation of Iron," by B.D. Cahan, C.T. Chen, D.J. Wheeler and F. Yeager, OMR Symposium on Interfaces (in conjunction with the West German-W.S. Mavy Scientific Data Exchange Agreement), Mational Bureau of Standards, Gaithersburg, MD, 13-20 October 1978.

- "Surface Properties of Flectrodes and their Relation to Electrochemical Reaction Rates," E. Yeager, Gordon Research Conference on Electrochemistry, Santa Barbara, Calif. 3-12 January 1979.
- 20. "Sample Transfer Techniques for Analysis of Electrode Surfaces by LEED-Auger Electron Spectroscopy," P. Hagans, A. Homa, and E. Yeager, Pittsburgh Analytical Meeting, Cleveland, 1-6 March 1979. (Abstract 448).
- 21. "LEFD-Auger-Thin Layer Electrochemical Studies of the Undernotential Deposition of Ph on Au Single Crystals," P. Hagans, A. Homa, and E. Yeager, Third Symposium on Electrode Processes, 'lational Heeting, The Electrochemical Society, Boston, 6-11 May 1979.
- 22. "Optical and Electrochemical Studies of Edsorbed Transition Metal Complexes and Their De Electrocatalytic Properties," <u>E. Yeager</u>, J. Zagal, B.Z. Mikolic, and R.R. Adzic, invited lecture, <u>ibid</u>.
- 23. "Electrocatalysis and Its Application in Electrochemical Energy Conversion," E. Yeager, Case Mostern Reserve University Chemistry Department Frontiers Lecture Series, April 25, 1970.
- 24. "Oxygen Electrodes for Electrochemical Energy Storage Systems," by E. Yeader, First International Assembly on Energy Storage, Mational Academy of Sciences (USA) and Council of Academies of Science (Yugoslavia), Dubrovnik, 27 May 1 June 1979.
- 25. "Non-traditional Approaches to the Study of Solid-Electrolyte Interfaces: An Overview," E. Yeager, invited opening lecture, International Symposium on Non-Traditional Approaches in Electrochemistry, "Snowmass, Colorado, September 23-27, 1979.
- 26. "O2 Electrolysis and its Application to Energy Conservation," <u>E. Yeager</u>, meeting of the Capitol Section of The Electrochemical Society, Washington, DC, March 6, 1980.
- 27. "Fundamental Aspects of 02 Electrochemistry", E. Yeager, University of Osaka, Japan, April 1980.
- 28. "Undernotential Electrodeposition", <u>E. Yeager</u>, at Amanashi University, Japan, April 1980.
- 29. "Fundamental Aspects of 02 Electrocatalysis," E. Yeaner, at Institute of Catalyst Research, Hokkaido University, Janan, April 1980.
- 30. "Adsorbed Transition Metal Macrocyclic Complexes on Graphite and Metal Surfaces: Optical and Electrocatalytic Properties," E. Yeager, U.S.-France Joint Seminar on Adsorptive Attachment and Chemical Bonding to Electrode Surfaces to Catalyze Electrochemical Reactions, N.S.F.-C.N.R.S., Isle of Bendor, France, June 22-25, 1980

- 31. "The Chemi-Conductor: A New Model for the Passive Film on Iron," by B.D. Cahan, 31st International Meeting of the International Society of Electrochemistry, Menice, September 22-26, 1980.
- 32. "Underpotential Deposition of Lead on Single Crystal Gold: LEED-Auger Thin Layer Cell Electrochemical Studies," by P. Hagans, A. Homa, W. O'Grady and E. Yeager, ibid.
- 33. "Ontical Reflectance Spectroscopy and Electrochemical Properties of Adsorbed Monolayers of Phthalocyanines," by B.Z. Mikolic, R.R. Adzic, J. Zagal and E. Yeager, ibid.
- 34. "Oxygen Electrodes for Industrial Electrolysis and Electrochemical Power Generation," E. Yeager, Case Centennial Symposium on Electrochemistry in Industry--Mew Directions, Cleveland, October 20-22, 1989.
- 35. "Recent Advances in the Science of Electrocatalysis," <u>E. Yeader</u>, address on receiving the Edward Goodrich Rolleson Medal, The Electrochemical Society Fall Meeting, Hollywood, Florida, October 7, 1980.
- 36. "Raman Studies of the Vibrational Properties of Adsorbed Species and Films at Electrochemical Interfaces," <u>E. Yeager</u>, R. Kotz and G. Nazri, The Electrochemical Society Spring National Meeting, Minneapolis, May 10-15, 1981.
- 37. "Electrocatalysis: Fundamental Aspects and Recent Developments," E. Yeager and D. Scherson, <u>ibid</u>.
- 38. "Auger and SIMS Studies of the Passivation of Iron-Chromium Alloys,"

  <u>S. Tjong</u>, J. Eldridge, R. Hoffman, R. Hehemann, and E. Yeager, The
  Electrochemical Society Fall National Meeting, Denver, Oct. 11-16, 1981.
- 39. "UHV-LEED-AES Techniques Applied to Single Crystal Electrochemical Interfaces," A. Homa, B. D. Cahan and <u>E. Yeager</u>, <u>ibid</u>.
- 40. "Spectroelectrochemical Methods," E. Yeager, Lecture at Whiteshell Nuclear Research Establishment, Pinawa, Canada, July 16, 1982.
- 41. "Surface Chemistry of Electrochemical Interfaces," <u>E. Yeager</u>, Langmuir Lecture, American Chemical Society National Meeting, Kansas City, Sept. 12-16, 1982.
- 42. "LEED-AES-Thin Layer Electrochemical Studies of Hydrogen Adsorbed on Platinum Single Crystals," A. Homa, <u>E. Yeager</u> and B. Cahan, International Conference on Electronic and Molecular Structure of Electrode-Electrolyte Interfaces, Logan, Utah, July 25-30, 1982.
- 43. "Study of Phthalocyanines in Aqueous Solutions and Adsorbed on Electrode Surfaces," B. Simic, S. Zecevic and E. Yeager, ibid.
- 44. "Applications of <u>in situ</u> Mossbauer Effect Spectroscopy to the Study of Electrode-Electrolyte Interfaces," <u>D. Scherson</u>, S. Yao, E. Yeager, J. Eldridge, M. Kordesch and R. Hoffman, <u>ibid</u>.

- 45. "Mechanistic Aspects of O, Electrocatalysis," by E. Yeager, D. Scherson and B. Simic-Glavaski, The Electrochemical Society National Meeting, San Francisco, May 1983. Extended Abstr. 83-1, 706 (submitted for publication in Proceedings of the Symposium on Electrocatalysis).
- 46. "Oxygen Reduction Electrocatalysis: Fundamental Aspects," by E. Yeager, The Electrochemical Society, National Meeting, Washington, D.C. October 1983. Extended Abstr. 83-2, 387.
- 47. "Oxygen Interaction with Adsorbed Tatrasulfonated Phthalocyanines," by S. Zecevic, B. Simic-Glavaski and E. Yeager, loc. cit. Extended Abstr. 83-2, 482.

### CONTRACT PERSONNEL

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- B. D. Cahan, Professor of Chemistry: Co-principal investigator (1976-83)
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  - 1. C. Chen (1977-78) Ph.D 1979
  - 2. Arthur Homa (1978-82) Ph.D 1982
  - 3. Michael Hanson (1980-84) Ph.D thesis completion Oct. 1984
  - R. Allen Wilkinson (1981-84) Ph.D thesis completion, spring 1985
  - G. McDougall (1982-84) Ph.D thesis completion, late 1985
- C. Postdoctoral appointments
  - 1. R. K. Sen (1976)
  - W. O'Grady (1976)
     B. Nikolic (1978)

  - 4. B. Simic-Glavaski (1977)

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